

Wavenumber Standards for Near-infrared Spectrometry

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1 INTRODUCTION

The near-infrared (NIR) spectra (15 000–4000 cm^{-1} , 666–2500 nm) of organic compounds are composed of overtone and combination bands of the fundamental vibrational transitions found in the infrared ($\approx 3000 \text{ cm}^{-1}$). The absorptivities of these NIR bands are 10–10 000 times lower than those found in the infrared. The combination of relatively weak optical absorption of these wavelengths, and therefore the probability of characterizing relatively thick samples, and the NIR transparency of glass and optical glass fibers enables the direct measurement of the NIR spectra of many materials – often with little or no sample preparation. This, more than anything else, has led to the explosive growth of NIR applications in agricultural, chemical, and pharmaceutical applications.

Traditional spectroscopic methods in analytical chemistry often relate the concentration of a molecular species to the intensity of just one of its absorption bands. Such univariate calibration methods are seldom useful in the NIR, where bands are broad and highly overlapped. Full-spectrum “chemometric” models are required to extract quantitatively the desired analyte concentrations from NIR spectra.¹

Successful multivariate calibration requires consistent representation of the relevant information in both the dependent and independent variables. For spectroscopic applications, consistent chemical concentration determination requires stability in the spectrophotometer’s wavelength/wavenumber (x -axis) as well as along its response (y -axis).^{2,3} Critical applications may require x -axis stability

on the order of a part in 10^4 to 10^5 over the time-course of a given measurement (calibration and prediction). Both dispersive and Fourier transform (FT) instruments are capable of this degree of x -axis stability, given appropriate maintenance and environmental control. However, demonstrating that a *given* instrument has the desired stability requires some form of x -axis calibration.

The development of wavelength/wavenumber calibration standards for NIR instruments poses several challenges. First, the spectral resolution of today’s NIR instrumentation ranges from fairly high (4 cm^{-1} , $\approx 0.4 \text{ nm}$) on FT instruments to fairly coarse (10 to 20 nm) for diode-array, filter, or rapid-scanning wavelength dispersive instruments. A wavelength standard applicable to the range of NIR spectrophotometric instrumentation with high repeatability and low bias would be desirable. This requires a standard with band features that do not change appreciably as a function of resolution. Secondly, NIR applications may require uniform spectral coverage from 14 000 to 5000 cm^{-1} (700 to 2500 nm). Most materials cannot offer this breadth of coverage using a fixed optical thickness. Thirdly, many NIR applications are based upon diffuse reflection, so wavelength standards should accommodate this sampling geometry and be applicable in diffuse reflection as well as transmission applications. NIR wavelength standards are therefore a compromise of spectral coverage and resolution and are targeted for a particular NIR application. The three major classes of NIR wavelength standard include: high resolution gas standards, custom glasses/crystals with f-shell electronic transitions, and single thickness organic standards that have well defined bands in one or more regions of the NIR spectrum. Only absorption/reflection standards

that can be placed at the sampling optic of the spectrometer will be considered here, as these are the only standards that can be used for full instrument validation. Compositional standards, i.e. those used to train sample-specific NIR applications (standard wheat kernels, grain samples, etc.), are outside the scope of this article.

2 WATER VAPOR

Atmospheric water is ubiquitous, and its rotational–vibrational bands occur throughout the NIR spectral region, most prominently between 5000 cm^{-1} and 5600 cm^{-1} , and 7000 cm^{-1} and 7425 cm^{-1} . Water lines are currently used by several spectrometer manufacturers for single-point calibration of the wavenumber axis of FT spectrometers.⁴ For the most part, the lines are too narrow and too closely spaced to be of practical use as wavelength standards for dispersive NIR instruments. Even at 0.125 cm^{-1} resolution, the highest resolution achievable with typical laboratory-based FT spectrometers, many water bands are actually unresolved multiplets. For example, the 7306.74 cm^{-1} line is commonly used for calibration, even though it is actually a closely spaced doublet.⁵ As a result, such band positions are dependent upon the instrument lineshape, band intensities, and the single-beam background absorption. The water line positions and assignments are tabulated in the HITRAN database⁶ as well as several references including the compilation of Guelachvili and Rao.⁷

3 GAS STANDARDS

The National Institute of Standards and Technology (NIST) provides several gas Standard Reference Materials® (SRMs®) and Standard Reference Databases that are useful for calibration of high-resolution NIR instruments. SRM® 2517 is an acetylene (C_2H_2) gas-filled, optical-fiber-coupled absorption cell intended for the calibration of wavelength division multiplexers used in optical fiber communications from 1513 to 1541 nm.⁸ The peak locations of 50 absorption lines of the R and P branches of the $\nu_1 + \nu_3$ rotational–vibrational bands of acetylene are certified with an expanded uncertainty of 0.0006 nm (0.003 cm^{-1}). The recently developed SRM® 2519 is similar to SRM® 2517, but with $\text{H}^{13}\text{C}^{14}\text{N}$ used as the wavelength calibrant.⁹ Fifty-one lines from 1528 to 1563 nm are certified, 21 with an expanded uncertainty of 0.0006 nm and the rest with an expanded uncertainty of 0.003 nm. The spacing of the bands of these gases is such that they are fully resolved at spectrometer resolutions of 0.25 cm^{-1} and higher. While these SRMs® provide traceability to the fundamental SI unit

(meter), the very narrow x -axis range of certified values limits their use for instruments that cannot utilize single-point calibrations.

4 POLYSTYRENE

Polystyrene, which is commonly used as a wavelength standard in the infrared, has been proposed for similar use in the NIR.¹⁰ As the absorptivities of the carbon–hydrogen combination and overtone bands decrease significantly toward shorter wavelengths, a single thickness of polystyrene, or any other organic material, is unlikely to provide features of adequate intensity to cover the entire NIR wavelength range. Further, because the NIR band shapes of polystyrene are highly asymmetric, the band locations are a strong function of spectral resolution. Accordingly, NIST’s 38- μm -thick polystyrene SRM® 1921a Infrared Transmission Wavelength Standard¹¹ may be useful for some NIR applications, but it is not suited as a general NIR x -axis validation standard.

A number of spectrometer vendors, motivated by polystyrene’s low cost and availability, are currently using 1- to 2-mm thick polystyrene sheets as an x -axis validation standard for both FT and dispersive NIR instruments. These thicker samples have not been certified for use in the NIR, although several instrument makers have “piggy backed” the calibration of thicker polystyrene samples with overlapping certified bands of SRM® 1921 in attempts to produce a traceable NIR wavelength standard.¹² While this approach may be serviceable for instruments that may be calibrated with a single point, extension of this practice to dispersive instruments is not valid.

Polystyrene has several advantages as an NIR wavelength standard. As seen in Figure 1, its spectrum possesses reasonably sharp, but asymmetric, bands in the NIR. These bands have been found to be very reproducible for materials from widely different sources. Recent studies¹³ at NIST using polystyrene from four suppliers showed the average peak maximum difference to be less than 0.2 cm^{-1} ($\approx 0.1\text{ nm}$) for eight bands between 4000 cm^{-1} and 800 cm^{-1} . Source-specific differences were noted in the region above 5000 cm^{-1} where the bands were weaker and more asymmetric. Use of a centroid location algorithm (vide infra) can increase the precision of x -axis location measurements for such bands. Because the bands of polystyrene are highly asymmetric, however, the band positions are strongly dependent upon spectral resolution. As a result of the widespread appeal and use of polystyrene, both NIST and National Physical Laboratory (NPL), UK, have provided calibration services for customer-supplied lots of this material.

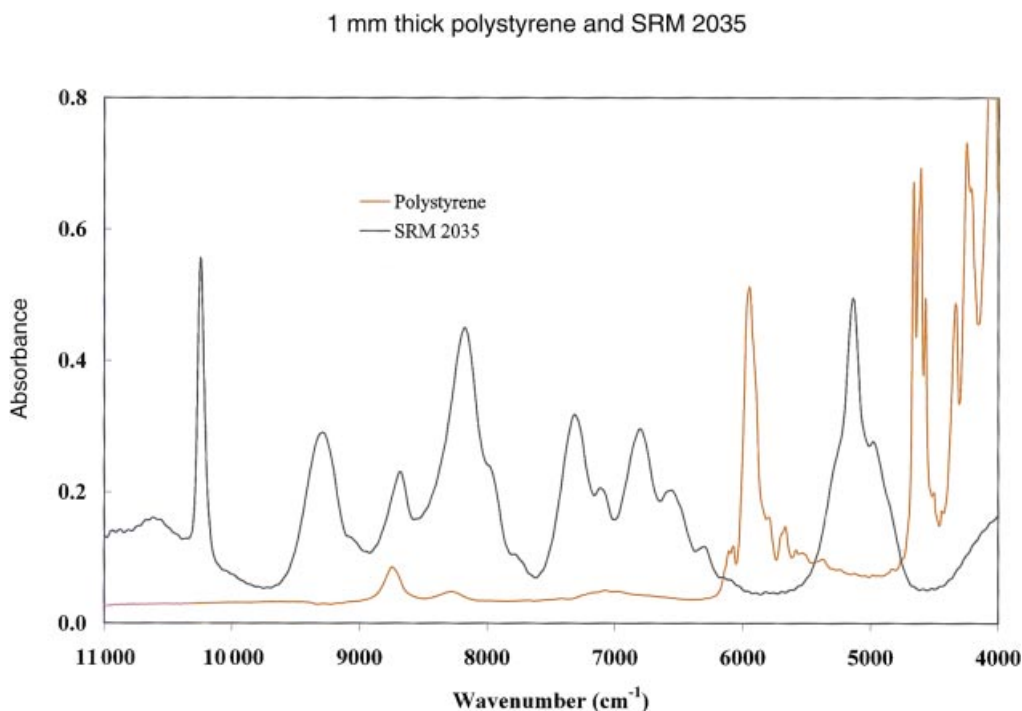


Figure 1. NIR spectra of polystyrene and SRM[®] 2035.

5 OTHER ORGANICS

Other organic materials used as standards include trichlorobenzene, which is described in the ASTM (the American Society for Testing and Materials) method E275-93.¹⁴ As a neat liquid, trichlorobenzene has reasonably sharp and well distributed bands, as do many chlorinated hydrocarbons, throughout the NIR spectral region. Although its spectrum is easily acquired in 1-cm pathlength cuvettes, industry resistance to solution-based standards precludes its wide acceptance. Solution standards, however, have the advantage of being inherent standards, in that for readily obtainable, high-purity materials, only data are required from a National Measurement Institute (NMI), not material. Polymers such as polystyrene, or poly(ethylene terephthalate), are easily machined, nonvolatile, and nontoxic, and can be easily incorporated into internal filter wheel accessories in NIR instruments for convenient calibration and validation of both the wavelength and photometric axis. For these reasons, solution-based standards are not expected to play a large role in the day-to-day calibration of NIR instrumentation.

6 RARE EARTH OXIDES

The NIR spectral characteristics of rare earth oxides (REOs) are well known.¹⁵ REOs form stable glasses in a number of

host matrices. For example, rare earths such as Nd³⁺ can be doped into yttrium aluminum garnet (YAG) and other crystals to form the basis of many of the telecommunications lasers used today. REOs were first used as NIR wavelength standards in the NIST SRM[®] 1920. The current version, SRM[®] 1920a, is a compacted mixture of erbium oxide (Er₂O₃), dysprosium oxide (Dy₂O₃), and holmium oxide (Ho₂O₃). This standard is designed for the calibration of diffuse reflection spectrometers operating over the range of 740 to 2000 nm.¹⁶ Thirty-seven reflection minima are certified in this region with an expanded uncertainty of 1 nm; however, only eight of these minima have sufficient spectral intensity to allow certification of the peak position over the range of spectral bandwidths from 2 to 10 nm. As the original intention of this SRM[®] was to calibrate dispersive reflection accessories, the certification did not include resolutions higher than 2 nm. Figure 2 shows the spectrum acquired at a moderately low resolution (64 cm⁻¹, ≈5 nm) overlaid with a more highly resolved (4 cm⁻¹, ≈0.4 nm) spectrum. Because neat powders are used, the sharp spectral features of the REO are conserved. As a result, the peak location is a very strong function of resolution. Efforts are underway to extend certification of SRM[®] 1920a to higher spectral resolutions.

There are a number of commercial versions of SRM[®] 1920. While most are “clones”, one vendor (Labsphere, Nashua, NH) has mixed each of the single rare-earth oxides in a powdered poly(tetrafluoroethylene) (PTFE) matrix and

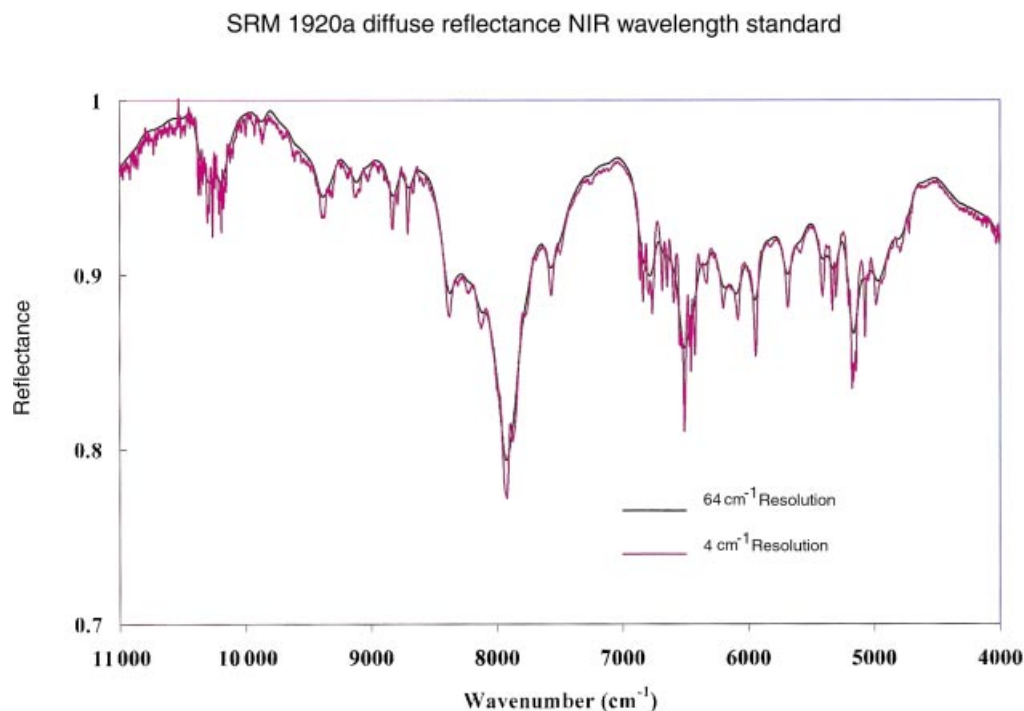


Figure 2. NIR spectra of SRM[®] 1920a at low (64 cm^{-1}) and high (4 cm^{-1}) resolution.

sintered the mixture together. This results in a robust diffusely reflecting wavelength standard that eliminates the specular reflection of the sapphire window used to contain the REOs in SRM[®] 1920. One caveat is that the sintering process appears to cause band shifts of one or more of the REOs and, therefore, the band locations are not precisely the same as those given in the SRM[®] 1920 certificate.

SRM[®] 1920 is only suitable for diffuse reflection measurements. REO transmission wavelength standards for the NIR have been available for some time through McCrone Scientific (London, UK). These are single REOs incorporated into a garnet matrix. Figures 3 and 4 illustrate the spectra of two of these proprietary wavelength standards (McCrone M-27 and Nelson M-42). Each exhibits very

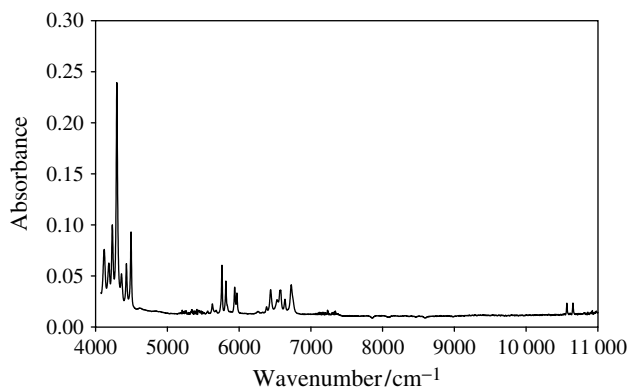


Figure 3. McCrone NIR wavelength standard.

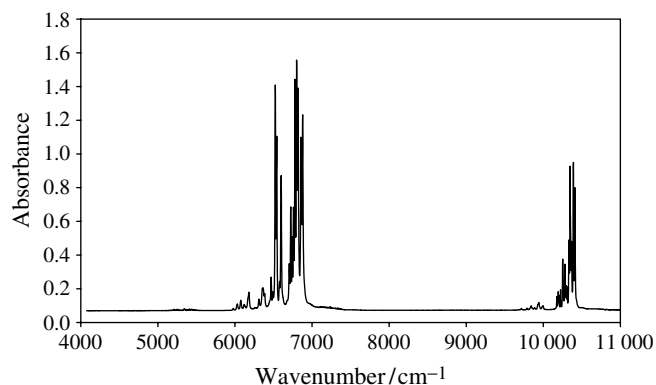


Figure 4. Nelson NIR wavelength standard.

sharp spectral features, but offers somewhat limited coverage in the NIR. Additionally, each crystal has bands in the UV–visible spectral region, making these standards very useful for the extended-spectral-range instruments on the market today. The sharp spectral features also provide a means of measuring instrumental spectral bandwidth. These standards suffer from the same drawbacks as SRM[®] 1920, in that the band locations are strong functions of spectral resolution, requiring certification of these bands at the appropriate bandpass for a specific instrument or application. NPL will certify these materials as wavelength standards. The proprietary formulation and limited availability of these materials prevent their widespread use as NIR standards.

Several other glass manufacturers have developed REO glasses primarily for optical filtering applications. Schott Glass Technologies (Duryea, PA, USA; Mainz, Germany) has developed a lanthanum sodium silicate host glass that can accommodate up to 30% by mass REOs. Eleven REOs have been singly incorporated into this matrix in the S-804x series filter glasses. The rare-earth doping in these glasses is too high to allow their use as wavelength standards, but these concentrations could conceivably be lowered and the REOs mixed to produce a useful NIR wavelength standard.

NIST started development of a NIR wavelength standard for transmission spectrometry in the fall of 1994 as a result of discussions with pharmaceutical manufacturers, NIR user groups, instrument vendors, the US Food and Drug Administration, and the Center for Process Analytical Chemistry at the University of Washington, Seattle. The desired design and performance specifications for this wavelength-axis validation standard were determined by evaluating existing NIR standards and their perceived limitations. The design and performance specifications of this new x -axis standard were: (1) wavelength coverage from at least 1000 to 2000 nm (10 000 to 5000 cm^{-1}); (2) peak location precision of at least 0.02 nm ($\approx 0.2 \text{ cm}^{-1}$); (3) wavelength bias (accuracy) of no more than 0.1 nm ($\approx 1 \text{ cm}^{-1}$); (4) fairly broad and symmetric peaks; and (5) ready availability. While more stringent than required by current regulatory practice,¹⁷ the location accuracy (precision and bias) specifications were chosen to enable chemometric methods development.² The peak width and shape requirements ensure that peak location is relatively insensitive to instrument resolution, permitting the use of the standard with instruments ranging from coarse to moderately high resolution.

These requirements led to the development of SRM[®] 2035, a wavenumber/wavelength standard for NIR transmission spectroscopy.¹⁸ A mixture of the oxides of samarium, ytterbium, and holmium (Sm_2O_3 , Yb_2O_3 , and Ho_2O_3) was identified that provides seven fairly symmetric and uniformly distributed bands from 976 to 1946 nm ($\approx 10\,250$ to 5139 cm^{-1}). The pre-melt composition includes 0.106 mass-fraction Ho_2O_3 , 0.042 mass-fraction Sm_2O_3 , and 0.025 mass-fraction Yb_2O_3 . These REOs are melted into a zirconia-stabilized, borate matrix suggested by Schott Glass Technologies, Inc. for its durability and processability. Figure 1 shows the NIR spectrum of SRM[®] 2035 recorded on a Bruker FT spectrometer overlaid with the spectrum of a 1 mm polystyrene sample for comparison.

SRM[®] 2035 is a batch-certified optical standard with a single set of certified values (band locations and associated 95% uncertainty limits). Batch certification considerably simplifies record keeping and minimizes the ultimate per-unit cost, but requires truly homogeneous materials and

careful quality assurance. The initial batch of 100 SRM[®] 2035 filters were all evaluated using a variety of dispersive and FT-NIR spectrometers to prove that all the bands have the same location in all the filters. Subsequent batches of SRM[®] 2035 (SRM[®] 2035a) have band location differences (compared with the original batch) on average of less than 1 cm^{-1} (0.2 nm) for the seven certified bands, which is not quite as reproducible as polystyrene from multiple sources, but remarkable for a complex glass. The use of a commercially available glass matrix and well known stoichiometry should ensure adequate supply of these reference materials.

Requiring peak location precision to be $0.02 \text{ nm} \approx 0.1 \text{ cm}^{-1}$ across measurement systems requires a very robust peak assessment algorithm. Of the many algorithms evaluated for this wavelength standard,¹⁹ only center-of-gravity (COG) methods²⁰ provided the desired high precision.

The COG or centroid method involves measuring the region around the band center in contrast to many peak maximum finding algorithms. The COG band location method used for SRM[®] 2035 was developed at NIST for the SRM[®] 1921 polystyrene infrared wavelength standard^{21,22} and is now adopted by the ASTM for certification of infrared wavelength standards (E1421-94).²³ The method as applied to SRM[®] 2035 and other NIST wavelength standards has been discussed previously.²⁴ COG methods have been criticized as defining a band feature that can significantly differ from the visual peak maximum, especially for asymmetrical peaks.²⁵ Reducing the fraction of the peak utilized in the algorithm, reduces the difference between the peak maximum and the centroid, but at the cost of decreased location precision. For SRM[®] 2035, a 10% peak area fraction was chosen empirically to provide the required band location precision, while also providing a band feature that matches the peak location typically found by conventional amplitude methods to within 1 cm^{-1} ($\approx 0.1 \text{ nm}$). Several methods are currently under investigation to provide the enhanced location precision of the centroid method, while minimizing the differences between the area and peak amplitude methods.²⁶ While easily implemented as a user-defined macro, COG methods are now beginning to be built into spectrometer vendors' software.

The SRM[®] 2035 certificate includes the certified values for the seven most prominent bands of this optical filter. The band locations are certified in both vacuum wavenumber and air wavelength at a number of discrete, constant wavenumber/wavelength resolutions. In addition, information values are provided for the peak locations in both wavenumber and wavelength using a peak amplitude method. The expanded uncertainty (95% confidence limit) for the band locations is on average less than

0.2 cm^{-1} ($\approx 0.02\text{ nm}$), for the spectral resolutions typically encountered in NIR applications. The certified band location values, supplementary information, and a description of the methods and calculations employed are provided in the SRM[®] 2035 certificate.

As many NIR applications may require spectrometer operation at temperatures different from those used at NIST to certify the band positions, the changes in band location with filter temperature have been accurately determined and are provided in the SRM[®] 2035 certificate. The temperature coefficients for all seven SRM[®] 2035 bands are less than $0.1\text{ cm}^{-1}\text{ }^{\circ}\text{C}^{-1}$ ($<0.02\text{ nm }^{\circ}\text{C}^{-1}$). The expanded uncertainty for the band locations listed in the SRM[®] 2035 certificate includes a $3\text{ }^{\circ}\text{C}$ range of filter temperature around a mean temperature of $25\text{ }^{\circ}\text{C}$.

The best results with this optical standard will be achieved by placing it within an enclosed sample position that is both temperature and humidity controlled. Many NIR applications, however, require calibration of an external accessory such as a fiber probe that is not in a purged and temperature-controlled environment. Future extensions of the certification of this standard could include a temperature compensation algorithm for the band locations. Also, many of the fiber probes in current use operate in trans-flection, where NIR radiation is reflected after traversing the sample and passed through the sample again before detection. To accommodate this mode of operation, NIST is certifying SRM[®] 2036, a quasi-diffuse reflection standard made by backing the SRM[®] 2035 glass with a piece of sintered PTFE.

Several options exist for the calibration of the wavenumber/wavelength axis NIR spectrometers. For chemometric applications of NIR spectroscopy, requiring validation of the x -axis, both polymer standards and SRM[®] 2035 have the necessary spectral coverage and precision to be useful. Polystyrene with its low cost and availability will find use as a built-in and day-to-day standard for single instruments. SRMs[®] 2035 and 2036 are more suited for use across the whole range of NIR instruments and as reference standards for those applications involving ISO (International Organization for Standardization) certification, multi-instrument and interlaboratory comparisons, good laboratory practices, and Food and Drug Administration regulated methods.

ACKNOWLEDGMENTS

Certain commercial equipment, instruments and materials are identified in this paper to specify the experimental procedure as completely as possible. In no case does such an identification imply a recommendation or endorsement by the National Institute of Standards and Technology, nor

does it imply that the material, instrument, or equipment is necessarily the best available for the purpose.

ABBREVIATIONS AND ACRONYMS

ASTM	American Society for Testing and Materials
COG	Center-of-gravity
ISO	International Organization for Standardization
NIST	National Institute of Standards and Technology
NMI	National Measurement Institute
NPL	National Physical Laboratory
PTFE	Poly(Tetrafluoroethylene)
REO	Rare Earth Oxide
SRM [®]	Standard Reference Material [®]
YAG	Yttrium Aluminum Garnet

REFERENCES

1. H. Martens and T. Naes, 'Multivariate Calibration', John Wiley & Sons, New York (1989).
2. K. Faber and B.R. Kowalski, *J. Chemometr.*, **11**(3), 181 (1997).
3. J. Workman and J. Coates, *Spectroscopy*, **8**(9), 38 (1993).
4. P.R. Griffiths and J.A. de Haseth, 'Fourier Transform Infrared Spectrometry', John Wiley & Sons, New York (1986).
5. R.A. Toth, *Appl. Opt.*, **33**(21), 4851 (1994).
6. L.S. Rothman, C.P. Rinsland, A. Goldman, S.T. Massie, D.P. Edwards, J.M. Flaud, A. Perrin, C. Camy-Peyret, V. Dana, J.-Y. Mandin, J. Schroeder, A. McCann, R.R. Gamache, R.B. Wattson, K. Yoshino, K.V. Chance, K.W. Jucks, L.R. Brown, V. Nemtchinov and P. Varanasi, *J. Quantitative Spectrosc. Radiat. Transfer*, **60**, 665 (1998); Phillips Laboratory/Geophysics Directorate, Hanscom AFB, MA (<http://www.HITRAN.com>).
7. G. Guelachvili and K.N. Rao, 'Handbook of Infrared Standards II', Academic Press, San Diego, CA (1993).
8. SRM 2517 Certificate, National Institute of Standards and Technology, 'Standard Reference Material 2517, Wavelength Reference Absorption Cell-Acetylene ($^{12}\text{C}_2\text{H}_2$)', Standard Reference Materials Program, NIST, Gaithersburg, MD, USA (<http://ois.nist.gov/srmcatalog/certificates/viewcert2.cfm?certificate=2517>).
9. SRM 2519 Certificate, National Institute of Standards and Technology, 'Standard Reference Material 2519, Wavelength Reference Absorption Cell-($\text{H}^{13}\text{C}^{14}\text{N}$)', Standard Reference Materials Program, NIST, Gaithersburg, MD, USA (<http://ois.nist.gov/srmcatalog/certificates/viewcert2.cfm?certificate=2519>).
10. K. Norris, personal communication (1992).
11. SRM 1921a Certificate, National Institute of Standards and Technology, 'Standard Reference Material 1921a, Infrared Transmission Wavelength Standard', Standard Reference Materials Program, NIST, Gaithersburg, MD, USA

- (http://ois.nist.gov/srmcatalog/certificates/view_cert2.cfm?certificate=1921a).
12. S.R. Lowry, J. Hyatt and W. McCarthy, *Appl. Spectrosc.*, **54**(3), 450 (2000).
 13. S.J. Choquette, 'Near-infrared Wavelength Characterization of Eight Absorbance Peaks of Polystyrene in the 10 000 cm^{-1} to 4000 cm^{-1} Region', Report of Analysis, 839.04-00-224, NIST, Gaithersburg, MD (2000).
 14. Annual Book of ASTM Standards, ASTM E 275-93, 'Standard Practice for Describing and Measuring the Performance of Ultraviolet, Visible, and Near-infrared Spectrophotometers', E 03.06, 723–733, ASTM, Philadelphia, PA (1999).
 15. G. Fuxi, 'Optical and Spectroscopic Properties of Glass', Springer-Verlag, New York (1992).
 16. SRM 1920a Certificate, National Institute of Standards and Technology, 'Standard Reference Material 1920a, Near Infrared Reflectance Wavelength Standard from 740 nm to 2000 nm', Standard Reference Materials Program, NIST, Gaithersburg, MD, USA (<http://ois.nist.gov/srmcatalog/certificates/viewcert2.cfm?certificate=1920a>).
 17. Pharmacopeial Previews, *Pharmacop. Forum*, **24**(4), 6463 (1998).
 18. SRM 2035 Certificate, National Institute of Standards and Technology, 'Standard Reference Material 2035, Near Infrared Transmission Wavelength Standard from 10 300 cm^{-1} to 5130 cm^{-1} ', Standard Reference Materials Program, NIST, Gaithersburg, MD, USA (http://ois.nist.gov/srmcatalog/certificates/view_cert2.cfm?certificate=2035).
 19. S.J. Choquette, J.C. Travis and D.L. Duewer, *Proc. SPIE*, **3425**, 94 (1998).
 20. D.G. Cameron, J.K. Kauppinen, D.J. Moffat and H.H. Mantsch, *Appl. Spectrosc.*, **36**, 245 (1982).
 21. D. Gupta, L. Wang, L.M. Hanssen, J.J. Hsai and R.V. Datla, 'Polystyrene Films for Calibrating the Wavelength Scale of Infrared Spectrophotometers (SRM 1921)', NIST special publication 260-122, PB95-226866/AS (1995).
 22. C. Zhu and L.M. Hanssen, 'Studies of a Polystyrene Wavenumber Standard for Infrared Spectrometry', in "The 11th International Conference of Fourier Transform Spectrometry", ed. J.A. de Haseth, AIP Conference Proceedings 430, American Institute of Physics, New York, 491–494 (1998).
 23. Annual Book of ASTM Standards, ASTM E1421-94, "Standard Practice for Describing and Measuring the Performance of Fourier Transform (FT-IR) Spectrometers: Level Zero and Level One Tests", ASTM, Philadelphia, PA, 818–822, Vol. E 03.06 (1999).
 24. S.J. Choquette, J.C. Travis, L.E. O'Neal, C. Zhu and D.L. Duewer, *Spectroscopy*, **16**(4), 14 (2000).
 25. R.A. Spragg and M. Billingham, *Spectroscopy*, **10**(1), 41 (1995).
 26. C. Zhu and L.M. Hanssen, *Proc. SPIE*, **4103** (2000).